UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

Sample Preparation Techniques for Transmission Electron Microscopy of Geologic Materials

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1. Reston, Va.

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1. Introduction

The Transmission Electron Microscope has been used in geologic research since the late 1940's, gaining in popularity as its potential and ease of use has increased. However, in order to transmit electrons through geologic material, the material must be very thin, usually less than 0.5 micrometers. These very thin sample preparations are called thin foils after the early metallurgical samples that were prepared by beating gold into a very thin foil. The problems associated with thin-foil specimen preparation of geologic materials are compounded, as they are brittle and commonly severely deformed. The body of literature concerning sample preparation is extensive, including the texts of Kay (1965), Hirsch and others (1965) and Glauert (1972). These deal most often with biological, metallurgical or ceramic samples and much less with geologic samples. This report discusses the problems associated with geologic sample preparation and describes methods that are routinely successful. The ideal thin-foil specimen is (1) electron transparent (0.5 µm for rockforming silicates examined in our JEOL 200B operating at 200 keV), is (2) a good representative of the bulk sample, and is (3) minimally damaged by the preparation technique. Any method used must fulfill these three requirements as well as possible. At the U.S. Geological Survey Reston TEM laboratory, we have refined some old techniques and developed new ones that fill the above requirements for a large variety of geologic samples with very good reproducibility.

Sample preparation is the most critical part of any TEM study. The object is to produce a doubly polished thin-section that can be successfully thinned using a standard ion-milling apparatus. Everything that is done during sample preparation is designed to maximize your chances for success at ion-thinning.

Samples with the greatest chance for success are doubly-polished with all pits and large scratches removed, ultra-thin (mechanically thinned to 10 µm or less), show minimum damage from the mechanical techniques used (ie. no cracks are induced during grinding and polishing steps) and are free of residual bonding materials left on the surface. A sample that meets these criteria can usually be ion-thinned.

A difficulity with geologic samples is that they are often not single crystal specimens, but are polycrystalline, polyphase aggregates. Each grain in the sample will behave differently during preparation. It is this problem that makes routine sample preparation difficult for the geologist. There are no guarantees that the same procedure will work for every sample. Specimen preparation is a combination of art and science and an intuitive feel for how a sample will react to a given technique. The Reston TEM laboratory procedures will work for standard thin-sections and are easily modified for difficult samples. Most of the samples done in our lab do not begin as the usual doubly-polished standard sized thin-sections. Frequently our samples are very small (<100 $\,\mu\text{m}$) very friable (like a lunar breccia), the one-of-a-kind variety or very soft (like a coal). We have developed general methods to encompass all of these non-standard geologic samples, though no two samples will react the same--even those from the same parent rock.

2. Thin-Section Preparation Methods

The Doubly-Polished Thin Section

Any sample used for Electron Microprobe analysis, SEM (for semi-quantitative analysis) and TEM requires a polished surface with a minimum of pits and scratches that will interfere with the Argon-ion beam of the ion-milling apparatus or the electron beam of the Probe, SEM and TEM. For any study that is to include TEM analysis a doubly-polished thin-section will need to be prepared.

The standard thin-section is $\sim 30.0~\mu m$ thick and 3.0 cm in length, is usually mounted on a slide using epoxy and is most often not polished but covered with a glass cover slip (for optical microscopy). This thin-section cannot be used for TEM analysis because it is not polished and cannot be removed from the slide. A doubly-polished thin-section mounted with a soluble adhesive is needed. A soluble adhesive is used because the sample must be ion-thinned on both sides and mounted in the TEM with no supporting glass slide. To prepare a doubly-polished thin-section follow this procedure:

- 1. Extract a small sample from the parent rock by sawing or chipping off a piece. The sample should be no larger than a $1\ 3/4\ x\ 1"$ rectangular slide or a 1" diameter round slide.
- 2. Cut the sample so that its dimensions are 1/8-1/4" less than those of the slide on all sides. It is best to use a round slide for very small or delicate samples as you have better manual control during the grinding and polishing stages.
- 3. Samples that are very friable or vesicular often need to be vacuum impregnated with epoxy to bind them together for better support during the grinding steps.

- 4. Clean and dust off a slide. (Clean with detergent followed by an ethyl alcohol rinse.)
- 5. Place the slide on a hot plate that is heated to approximately 135° C (use a surface thermometer and make sure that the hot plate is up to temperature before you start) and let it heat for one minute.
- 6. Drop on a small piece of Crystalbond and allow it to melt. Spread the Crystalbond evenly and thinly over the surface of the slide.
- 7. Place the sample into the Crystalbond and press down evenly on the sample to squeeze out any air bubbles underneath. Remove the slide from the plate and allow to cool.
- 8. We recommend the use of Crystalbond as an adhesive because it is acetone soluble and dissolves cleanly from the slide and sample. Canada balsam and Lakeside, which are used extensively for standard thin-sections are not suitable for TEM sections as they do not dissolve cleanly, but leave a fine gummy residue on the surface that will intefere with ion-thinning. Before you use Crystalbond, be sure that the hot plate is at 135° C. Excessive or prolonged heat will change its properties.

Preparation of a flat surface

- 1. Saw or grind flat the top of the sample until the largest area is exposed for polishing. If using a saw, grind off the saw marks using a 400 grit aluminum oxide slurry on a rotating lap. If you choose to grind a flat side also use the 400 grit slurry.
- 2. Ultrasonically clean the sample in tap water for a few seconds. Clean the grit from the wheel with tap water.
- 3. All aluminum oxides are prepared as slurries. Place 3 tablespoons of grit in a 250 ml plastic squirt bottle and fill 3/4 full with tap water.

Shake vigorously before each use. We do not recommend the use of silicon carbide grit as it produces too many cracks in the specimen.

- 4. Remove the 400 grit scratches by grinding with 600 grit for a few minutes.
- 5. Ultrasonically clean the sample in tap water then clean off the wheel.
- 6. Place the next grit size (800) on a flat glass surface, such as a large air photo plate, and grind by hand for a few minutes to remove the coarse scratches.
- 7. Ultrasonically clean, then dry the sample, then clean the glass.
- 8. Place the final grinding grit (1200) on the glass and grind by hand for a few minutes. Ultrasonically clean, then dry the sample.
- 9. If at any time during the grinding stages pluck-outs occur, discontinue using that grit size.
- 10. Clean off the plate.
- 11. Clean and dry the sample and check for damage-pluckouts and fractures that are induced during the grinding steps. Damage lessens the chance for successful ion-thinning. If the damage is minor go to the next lower grit size. Once damage has occurred do not use the rotating lap. A sample with grains that pluck out easily requires a very light touch if you are to successfully complete it. If, at the time of pluckouts, the sample is very thin go directly to the polishing steps.

Preparation of a polishing wheel

We have found that a silk cloth mounted tightly on a glass wheel is the best surface for polishing geologic samples.

1. On an 8" brass wheel covered with an 8" round of glass held in place with parafin, place a clean round of silk. Make sure that the silk is on tightly so there are no creases.

2. Shake the can of diamond spray thoroughly and spray a generous amount on the silk. This is best done on a rotating lap that is spinning slowly. We use diamond spray rather than paste because it is less messy and the diamonds are more evenly distributed on the silk.

Polishing a flat surface

After producing a flat top, the next step is to polish the surface. This is a time consuming trial-and-error process. Some samples will polish in a short time (a few minutes) while others will take a much longer time. Some samples will only polish with a certain diamond size while others will polish in the usual 9 μm to 1 μm diamond size sequence. Generally, single crystals will polish quickly while multiphase and multihardness samples will require more time.

- 1. Starting with 9 μm diamond spray, polish the sample using a circular or figure 8 motion for one minute.
- 2. Thoroughly clean the slide with distilled water and check for a beginning polish under a microscope, keeping a particular area in mind. Polish for another minute.
- 3. Continue checking the polish until there is no more improvement at that diamond size. A good 9 μm polish will show the removal of the larger pits and scratches from the surface and a mirror-like surface will begin to appear.
- 4. Place a clean silk cloth impregnated with 6 μm diamond spray on a glassbrass wheel. Again polish until there is no more improvement.
- 5. Continue to the 3 μm diamond spray and finally to the 1 μm diamond spray.
- 6. Between diamond sprays, clean the sample <u>thoroughly</u> in the ultrasonic cleaner with distilled water and liquid detergent to remove the diamond spray

oil.

- 7. At each polishing step you will be removing more of the pits and the larger scratches but you will also be putting light scratches on the surface. Each diamond spray polish will remove scratches from the previous diamond's polish.
- 8. The 1 μm scratches may be removed by polishing lightly with AB alpha or AB gamma polishing alumina on a microcloth covered wheel. Polish the sample on the alumina for 15 seconds.
- 9. Ultrasonically clean then dry the sample and check the final polish. A good polished surface is as free from all pits and scratches as possible and shows no induced fractures.

Removing the sample from the slide

- 1. If you are satisfied with your polished surface, remove the sample from the glass slide by soaking it in acetone until the sample floats off.
- 2. Clean the sample with a few drops of chloroform and dry.
- 3. Clean (use detergent followed by an ethyl alcohol rinse) and dust off a slide.
- 4. Place the slide on a hot plate that has been heated to approximatly 135° C (make sure that it is up to temperature before you start) and let it heat for several minutes.
- 5. Drop on a small piece of Crystalbond and allow it to melt. Spread the Crystalbond evenly and thinly over the surface of the slide.
- 6. Place your sample, polished side down, into the Crystalbond. Press the sample down evenly to squeeze out any air bubbles. Remove the slide from the plate and allow to cool.

Preparing the second surface

We now wish to make the sample standard thin-section thickness. If the

sample is strong and thick it may be sawed; if not, you will have to grind it to the correct thickness.

- 1. Starting with 600 grit on the wheel, grind to approximately 3rd order colors (refer to the Kerr text on mineralogy). The use of interference colors, as described by Kerr, is only useful for the transparent phases of minerals. Opaque minerals will not show interference colors. To judge the thickness of opaque mineral phases the use of a calibrated stage on an optical microscope is helpful. By focussing on the upper and lower surfaces of the sample, the thickness can be determined. The use of 400 grit is not recommended at this stage. Check the sample frequently for thickness and damage. Pay particular attention to the development of any fractures. If you have damaged the sample in any way, discontinue use of the rotating lap and complete the grinding gently by hand on the glass plate using a lower grit size.
- 2. When the sample is about 3rd order colors, use 800 grit on the glass plate to high 2nd order colors. Grind with a very gentle circular motion. If the sample is not uniformly thin, place fingers over the thicker area until the wedge is removed and the sample exhibits uniform interference colors.
- 3. Proceed to 1200 grit to remove any scratches or damage if any, that has occurred. As the sample gets thinner it is easier to cause damage such as deep scratches and pluckouts. It is essential that you lighten the pressure you are applying to the sample as it is thinning and that you immediately go to a smaller grit size if you have caused any damage.
- 4. Repeat the procedure for polishing the surface. <u>Do not</u> ultrasonically clean for more than 20 seconds as you may vibrate the sample from the slide or produce bubbles between the sample and the glass slide. Frequently check for polish, pluckouts and thinned-away areas during polishing. Remember to use a light touch because of the thinness of the sample.

The Ultra-Thin Specimen

The thin-section discussed in the previous section is the standard thickness of approximately 30 μm useful for optical microscopy, Electron Microprobe analysis and SEM work. However, for TEM work it is essential that the grinding and polishing stages produce a very thin (15 μm or less) sample and a relatively scratch-free surface. The thinner the sample is made in these stages the less time will be required during the ion-milling process, the less ion-beam damage will occur and, most importantly, greater amounts of electron transparent area will be produced.

- 1. Follow the procedure outlined in the section on <u>Doubly-Polished Thin Sections</u>. Pay particular attention to the polishing steps. Any pits and fractures left on the surface may cause irregular ion-beam milling. Light scratches are tolerable.
- 2. <u>Do not</u> ultrasonically clean after the final grinding stage unless the sample is particularily tough. Flush the sample thoroughly with distilled water as an alternative.
- 3. For TEM work, the sample should be ground down to between 5-15 μ m. The thinner the sample is, <u>without</u> damage, the better. Remember, the thinner the sample is, the more fragile it becomes. Use a light but even touch during all grinding and polishing steps. Because the sample must support itself during ion-milling, it is important not to introduce cracks into the specimen.
- 4. For TEM work the sample may now be removed from the slide by soaking the slide in acetone until the sample moves freely from the slide. Do not try to coax it from the slide with probe tools because you may cause it to break up. Sample integrity is very important at this stage.
- 5. Clean the sample with a few drops of chloroform to remove the last traces of the adhesive. Do this under a binocular microscope.

Microsized and Very Fragile Specimens Prepared for Ion-thinning

Small grains (30-1000 μ m) that will be ion thinned should be made as thin as possible mechanically (grinding and polishing). Preparing very small samples and very fragile specimens is similar to ultra-thin specimen preparation. The use of motorized grinders and polishers is not recommended at any stage as they will be too hard on the sample. A very light touch at all steps is required. And, the sample must be checked more frequently at all steps as changes occur very rapidly. Do not use the ultrasonic cleaner after the first side is polished.

- 1. It is better to imbed a very small or fragile sample in epoxy rather than Crystalbond. Many epoxy's are suitable for use but the best epoxy's are transparent, fast setting with short curing times and have a viscosity similar to syrup. We usually use DER 332 epoxy made by Dow Chemical. This epoxy mixes in a ratio of 5 parts epoxy to 1 part hardner (by volume) and must be precisely measured using disposable syringes. The epoxy will seep into friable samples to make them stronger and gives better support to very small samples.
- 2. Thoroughly clean and dry a slide and place the sample on it.
- 3. Place several drops of epoxy over the the sample, making sure that it is completely covered and epoxy gets underneath the sample. Spread the epoxy evenly on the slide to within 1/4" of the edge. This forms an epoxy "button" enclosing the sample. Allow the epoxy to set overnight then cure in a 100° C oven for 30 minutes the next day.
- 4. Grind the sample by hand as you would for a doubly-polished section.
- 5. Grind with 800 grit until the sample is about to be exposed through the epoxy.
- 6. At this point, go to the 1200 grit and proceed. For very small samples

- 1200 grit may be too harsh, therefore use 9 µm diamond to expose the surface.
- 7. Polish the sample the same way as for standard sections.
- 8. Using a micro-scalpel, gently pry the epoxy button from the slide. The epoxy will be very brittle, so be aware of cracking. If the epoxy is not coming off the slide easily, place the slide in a beaker of hot water for several minutes. This will soften the epoxy for easier removal. (If all else fails use the epoxy remover recommend for the type of epoxy used. This should be avoided as these removers are very toxic and the sample will almost certainly break into very small pieces.) After removing the epoxy button, trim off the ragged edges of the epoxy.
- 9. Clean a round glass slide and heat for one minute on a hot plate at 135° C. Place a piece of Crystalbond on the slide and allow to melt, then spread evenly.
- 10. Place the button polished-side down into the Crystalbond and press down gently to remove all air bubbles. Do not press directly over the sample.
- 11. Place a small piece of Crystalbond on top of the button and allow it to melt. Spread it evenly over the button and slide then remove the slide and cool.
- 12. Grind the sample with 800 grit just until the epoxy begins to show through the Crystalbond.
- 13. Grind with 1200 grit for a few minutes to expose the sample and remove the 800 grit scratches. Do not grind with 1200 grit for those grains that are on the order of 50 um or less.
- 14. When the grain begins to show through the epoxy go to the 9 μm diamond spray and polish off the remaining epoxy covering the grain.
- 15. Clean the sample under running water as it is probably too fragile for the ultrasonic cleaner. Do all steps very gently.
- 16. Polish the sample down to 1st order white or grey. Polish with a gentle

but even pressure. Check the surface frequently for polish and damage. If you are causing any damage, go to a lower diamond size and slow down.

18. When you have reached the desired thinness polish the sample gently with alumina on a Microcloth covered wheel for 1 minute to remove any 1 µm diamond scratches. You now have a finished microsized specimen.

19. The sample may be dissolved off with acetone for mounting on TEM grids. If possible, remove the sample from the surrounding epoxy before mounting. We have successfully made ultra-thin sections of grains as small as 30 μ m using this procedure.

Particulate and Grain Mounts Mounted Directly on TEM Grids

Often there is not enough material of an adequate size (at least 30 μ m) to make a single crystal mount, so a dispersion mount is made.

- 1. Begin by crushing a small number of grains to a fine powder with an agate mortar and pestle.
- 2. Place a micro-spatula tip full of the sample in a 10 ml specimen vial. Fill the vial 3/4 full with <u>double-distilled</u> water, ethyl alcohol or acetone. Use the ethyl alcohol if water will leach out some of the minerals in the sample.
 Acetone may be used for the same reason or if a fast evaporation is needed.
 (Do not use ethyl alcohol as the dispersing medium when using Collodian film covered grids, as the alcohol will dissolve the film.)
- 3. Place the sample vial in an ultrasonic cleaner and disperse the sample for several minutes. If the sample has a tendency to form clumps (clays, for example), break the clumps by using a carefully cleaned sonic probe immersed in the vial.
- 4. Using a micropipette, place a single drop of the sample on a 100 mesh grid covered with Holey carbon, Formvar or Collodian film and allow to dry. The

drying process will take approximately one hour if using water.

- 5. When the sample is dry, coat the surface with a light layer of carbon to prevent glow-discharge of the sample in the TEM.
- 6. Remove the sample grid from the slide by gently edging around the perimeter with fine-tipped tweezers to break the film bond. <u>Delicately</u> nudge the grid to release it from the slide. The sample grid can now be manipulated with the vacuum tweezers.
- 7. Assorted size grids covered by the above substrates are commercially available, but they are expensive. Collodian covered grids (used most often in the Reston TEM lab) can be easily made in the lab as follows:
- a. Place five drops of a collodion solution (1.0 % in Amyl Acetate) in a 5" diameter pyrex dish filled with double-distilled water.
- b. Allow the film to dry for several minutes. You will notice the spectrum of interference colors appear as the film dries. The colors correspond to the differences in thickness of the film.
- c. Place several grids in a square array with the shiney-side up on the pale yellow area of the film. Gently touch each of the grids to the surface of the film with the tips of a pair of fine tweezers to anchor them to the film.
- d. Cover a clean glass slide with the oil from your fingers to prevent the Collodian film from sticking to the slide which would inhibit its removal.
- e. Place the slide at a 45° angle to the film. Pushing the slide into the water, scoop up the grids so that the grids are lying on the slide covered by the film. Allow the grids to dry for at least one hour in a dust-free environment such as a dessicator.
 - f. Coat the grids with a light layer of carbon to make the film stronger.
- 8. If your sample is a single grain less than 30 μm it is best to crush the grain between two clean round glass slides.

- 9. Using a clean probe tool, gently place the small pieces of the sample directly onto a previously prepared grid.
- 10. Alternately, you can place a drop of liquid directly on the fragments and pass a coated grid through the liquid to collect the fragments. Allow the sample to dry as previously discussed.

Ultra-thin Sections of Coals

Sections of bituminous coals have been successfully prepared for TEM observation. Coal sections need to be mechanically thinned to 5 mm or less. If the coal has a high inorganic particle content it will not thin uniformly. It is best to use very small sections of the coal -no larger than 10 mm- as these are easier to manipulate and less likely to crack apart.

- 1. Follow the procedures outlined in the section on Microsized and Very Fragile Specimens but do not use any slurries coarser than 1200 grit. The epoxy and Crystalbond will have to be trimmed away from the slide as the sample thins or they will retard uniform thinning and scratch the coal surface.
- 2. When the sample is optically transparent and all optical microscopy and photographic work has been done, epoxy 100 mesh grids onto areas of intrest.
- 3. Holding on to the side of the grid using fine point tweezers, thoroughly scrape off any excess epoxy from the grids before placing on the coal to prevent it from pooling in the grid openings.
- 4. Allow the epoxy to dry overnight but do not cure the epoxy in an oven.
- 5. Remove the coal section from the slide by soaking it in acetone. The acetone will dissolve the Crystalbond from the slide and sample but will not dissolve the epoxyed grids from the coal. Any areas not covered by grids will likely break into small fragments.
- 6. Trim away excess coal not covered by the grid. Clean the epoxyed sections

with a few drops of chloroform.

3. Ion-beam Thinning

Following the initial thinning steps for all but dispersion mounts, the sample is still too thick for observation at 200 keV in our JEOL 200B microscope. Electron transparency for our instrument is 0.5 µm for common rock forming silicates. (This figure actually varies depending on the mean atomic number of the sample.) To achieve electron transparency, the specimen is further thinned by ion-bombardment. This is a process by which atoms or molecules are ejected from the sample by bombarding it with positive ions. The use of ion-bombardment for non-metallic specimens (discussed in Barber(1970) and Gillespie et al.(1971) was greatly advanced by the work of Palus and Reverchon (1962), whose advancements included the introduction of a rotating stage that allowed the sample to slowly rotate in two opposing ion beams at a predetermined angle, eroding both surfaces simultaneously and uniformly. The Gatan Model 600A in our lab is a typical micro ion-milling device found in many TEM labs. The glancing angle used for our work is 18°. A 6 kV ion potential is used for most samples. (It is best to use a 4 kV ion potential for coal samples as the heat produced by higher energies will often burn the coal.) Our positive ion source is Argon gas.

The Gatan Model 600A is equipped with low energy guns capable of removing the amorphous damage layer of sample material commonly formed on silicates that is caused by the high energy guns. The removal of beam damage is necessary for high resolution work. The preparation of very thin samples (10 μ m) during the mechanical thinning steps allows for a more efficient use of the ion-thinner by reducing thinning times and producing a larger area of electron transparency. Ultra-thin samples are mounted and ion-thinned using the following procedure:

4. Mounting The Sample

- 1. After a mechanically thinned sample has been removed from the slide, it must be mounted on a copper, gold, titanium or carbon grid or washer. Very small grains should be mounted on the grid bars of a 3 mm diameter titanium, copper or gold, 75, 100, 150 or 200 mesh grid. (The order of grid composition reflects the resistance to ion-thinning of the grid bars. The titanium grid bars will take the longest time to thin away.) If you are unable to mechanically thin the sample to 15 μ m or less, greater times will be required in the ion-thinner and you will often thin the bars away before the sample is thinned. This will require the removal of the sample from that grid (if it has not fallen through) and placing it on a new grid.) Larger intact samples need only be glued to a slot or hole washer.
- 2. Sample size must not exceed the size of the washer-3 mm, or it will not fit in the TEM sample holders. If the area of interest is too large, a small portion of the specimen must be carefully removed by breaking it away from the rest of the thinned sample.
- 3. On a clean glass slide place a small drop of any brand of Alpha Cyanoacrylate adhesive (Superglue). We recommend the use of Aron Alpha CE-475 Ethyl Slow #202. This adhesive has a slow set-up time (20 sec.) that gives you the time needed to manipulate the grid. These "Superglues" are best stored in a cool, dry place such as a refrigerator. Stored unopened they will last for a year. Once the tube is punctured, the glue will remain viscous for six months. To remove un-set glue from any surface soak in acetone for several minutes.
- 4. To mount the grid to the surface of the sample prior to ion-thinning proceed as follows:
 - 1. Holding the edge of the grid with fine tweezers, dip the non-shiny

surface into the glue and scrape the excess onto the slide. It is important to remove as much of the glue as possible from the grid as anything but a thin coating will greatly increase the drying time and reduce the binding strength. (Superglue requires only a molecular layer for binding.)

- 2. Pick up the sample with the glued surface of the grid and allow to air dry for several minutes before placing grid-side down on a clean glass slide. Allow the sample to dry for one hour.
- 5. Using the vacuum tweezers, place the grid on one of the stages of the ion-thinner. The gun on the left hand side of the Gatan thinner will operate under normal and liquid nitrogen conditions and has a clamping sample stage for good thermal conductivity. However, it is not necessary to clamp the sample down when thinning at room temperature conditions. Thinning times will differ markedly with each sample. Silicates, for example, may require much longer times (10 to 20 hours) than coal which may thin in as little as 15 minutes.
- 6. Set the manual timer for no more than 5 hours at a time. Check the sample for signs of thinning every 1/2 hour.
- 7. When the sample begins to show signs of thinning check every 10 minutes or less. Once a hole has appeared, thinning may be stopped or be allowed to continue to increase the thinned area. Keep in mind that once a hole has appeared the sample will continue to thin at a very fast rate. It is best to know the components of the sample to be thinned to best judge the time and how closely it needs to be watched.
- 8. When the sample has been thinned to your satisfaction, remove it from the holder and coat it with a thin layer of carbon. (The carbon layer need be only a few tens of Angstroms, not several hundred Angstroms as required for the Microprobe.)

5. Discussion

Sample preparation for Transmission Electron Microscopy has become more of a science in recent years with the use of more sophisticated automatic grinders and polishers. However, these machines are designed mainly for metal and ceramic samples and are often too brutal for many geologic samples. They are effective when making standard 30 µm thin-sections, but it is not recommended that they be used in the preparation of most TEM samples. Without the use of these automatic machines, TEM sample preparation is a time-consuming job and requires patience and experience. It is best to begin practicing with samples that are strong and abundant and show little alteration (altered grains are very fragile and pluckouts are quite common). Single crystal specimens are good to start with as they usually thin uniformly and polish quite readily. It is also best to start by making standard thin sections to get a feel for the techniques used in ultra-thin section preparation. Once the basic techniques have been mastered, greater success at sample preparation is gained by developing a feel for which sample techniques will cause the least amount of damage to your sample and produce a good sample in a reasonable amount of time.

6. List of Consumable Manufactureres

Crystalbond may be obtained from: Aremco Products

Box 429 Snowden Ave. Ossining, NY 10510

Buehler polishing products may be obtained from:

Buehler Ltd.

2120 Greenwood St. P.O. Box 1459 Evanston, IL 60204

Quantity	<u>Item</u>	Catalog No.
1 lb.	400 grit Al ₂ 0 ₃ 600 grit Al ₂ 0 ₃ 800 grit Al ₂ 0 ₃ (22.5 micron) 1200 grit Al ₂ 0 ₃	40-6425-400-016 40-6430-600-016 40-6622-225-016 40-6614-145-016
	(14.5 micron)	40-0014-143-010
1 qt.	Ultramet Sonic Cleaning Soln.	75-5000-032
5 oz.	1 micron diamond spray 3 micron diamond spray 6 micron diamond spray 9 micron diamond spray	40-6264 40-6268 40-6272 40-6276
10	Microcloth 8" polishing cloths	40-7218
10	Selected silk, 10" for 8" wheel	40-7408
6 oz.	AB alpha polishing alumina Linde A	40-6352-006
6 oz.	AB gamma polishing alumina Linde B	40-6353-006

Aron Alpha CE-475 Ethyl Slow #202 Superglue may be obtained from: E.T. Horsey and Co. 31005 Solon Rd.

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